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**COMPATIBILITY OF POLYVINYL CHLORIDE FILTER BAGS
WITH CHLOROFORM VAPORS**

Christopher J. Karwacki

RESEARCH AND TECHNOLOGY DIRECTORATE

May 1997

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AUTHORS Christopher J. Karwacki

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13. ABSTRACT (Maximum 200 words) A determination is made on the compatibility of polyvinyl chloride (PVC) filter bags when exposed to chloroform vapors resulting from offgassing of contaminated adsorbent filters. The objectives of this investigation were to estimate the concentrations of chloroform desorbed from Rapid Response System (RRS) filters previously exposed to chloroform and evaluate the compatibility of PVC sheet to chloroform vapors. The results of the investigation showed that desorption of chloroform vapors from the RRS filter can result in concentrations of approximately 0.2 to 13.0 mg/L when sealed in a closed container. The amount of desorption is primarily dependent upon the initial loading of chloroform on the adsorbent and the temperature of the filter bed. The PVC compatibility results showed that the equilibrated weight uptake on PVC sheet was 33.6 $\mu\text{g}/\text{cm}^2$ when exposed to 10.0 mg/L vapor concentration. The results support the work of other investigators that showed significant uptake of chloroform on PVC in liquid immersion tests. Although no permeation of chloroform through PVC sheet was observed during the 10-day exposure periods, the measured uptake could indicate the initial stages of long term permeation.				
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PREFACE

The work described in this report was authorized under Project No. 10262622A553, CB Defense/General Investigation. This work was started in July 1995 and completed in September 1995. The experimental data are recorded in laboratory notebook 95-0057.

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CONTENTS

1.	INTRODUCTION	7
2.	PVC-CHLOROFORM COMPATIBILITY	7
3.	VAPOR EQUILIBRIA	8
4.	EXPERIMENTAL METHOD	9
4.1	Adsorption Isotherms	9
4.2	Desorption from Preloaded Adsorbent	10
4.3	PVC-Chloroform Compatibility	10
5.	RESULTS AND DISCUSSION	10
5.1	Chloroform Equilibria	10
5.2	PVC-Chloroform Compatibility	11
6.	CONCLUSIONS AND RECOMMENDATIONS	12
	LITERATURE CITED	19

FIGURES

1	Schematic of Adsorption Isotherm Apparatus	13
2	Schematic of Desorption Apparatus	13
3	Schematic of PVC-Chloroform Permeation Test Apparatus	14
4	Adsorption Isotherms of Chloroform and Coconut Carbon at 25 and 55 °C . . .	14
5	Desorption Isotherms from Samples of Coconut Carbon Preloaded with Chloroform at 25 and 55 °C	15
6	Uptake of Chloroform on PVC Sheet at 10.0 mg/L Vapor Concentration (Ambient Temperature: 25 °C)	15

TABLES

1	Estimate of Chloroform Headspace Concentrations in the RRS Filter (Chloroform Loading: 0.1 g/g, Temperature: 25 °C)	16
2	Estimate of Chloroform Headspace Concentrations in the RRS Filter (Chloroform Loading: 0.5 g/g, Temperature: 25 °C)	16
3	Estimate of Chloroform Headspace Concentrations in the RRS Filter (Chloroform Loading: 0.1 g/g, Temperature: 55 °C)	17
4	Estimate of Chloroform Headspace Concentrations in the RRS Filter (Chloroform Loading: 0.5 g/g, Temperature: 55 °C)	17

COMPATIBILITY OF POLYVINYL CHLORIDE FILTER BAGS WITH CHLOROFORM VAPORS

1. INTRODUCTION

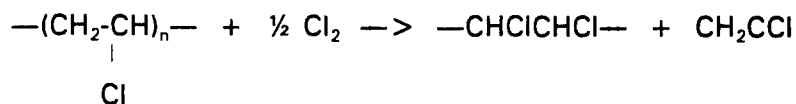
The handling and disposal of gas filters contaminated with hazardous compounds is an issue of increasing concern due to the nature of the adsorbed chemical if desorption occurred. The Rapid Response System (RRS) uses a four-stage filter unit consisting of two filters that contain a coconut grade activated carbon for removal of chloroform vapors, followed by two filters that contain NBC adsorbent for removal of CW agents. Typical operations may involve the decontamination of mustard, lewisite, phosgene, cyanogen chloride, chloropicrin, and simulants for nerve. In addition, chloroform and *tert*-butyl alcohol will be used as solvents in the decontamination process. Of all chemicals used, the greatest burden on the RRS filter may be chloroform, because it will be present in significant quantities and is highly volatile. Recent investigations have shown that chloroform is reversibly adsorbed and that significant desorption can occur.¹ Under dynamic flow, desorption can occur rapidly, but even under static conditions, such as a sealed filter, the propensity for desorption exists.

Safe handling procedures of filters contaminated with hazardous compounds have adopted the use of polyvinyl chloride (PVC) sheet to seal the filter to prevent a potential release of product. Even though the storage life of these filters may be as high as 90 days and that they are double bagged, the concern is that polymers that are incompatible with the hazardous compound may degrade and permeate the barrier. The objectives of this investigation were to estimate the concentrations of chloroform desorbed from Rapid Response System (RRS) filters previously exposed to chloroform and evaluate the compatibility of PVC sheet to chloroform vapors.

2. PVC-CHLOROFORM COMPATIBILITY

Polyvinyl chloride is used in many applications due to its ability to compound with many chemical additives, producing a wide range of physical and chemical properties. Some of the most important properties of PVC are flexure, abrasion, light stability, low vapor permeation, and high chemical resistance. Flexible PVC sheet by design is typically impermeable and is suited for the containment of many chemical compounds in solid, liquid, and vapor form.

The compatibility of PVC with a chemical is primarily dependent upon its resistance to chemical attack. Its principal vulnerability is free radical chlorination under the influence of UV radiation that can occur either at the C₁ or C₂ positions:



By design, an increase in the chlorine content may be desirable and can lead to increased mechanical strength and improved stability to light and heat. One feature of the chlorination reaction is swelling of the PVC film. In one process, the addition of a swelling agent using chloroform or carbon tetrachloride is advantageous but can be detrimental to many PVC products.^{2,3,4} The concern with the swelling of PVC is significant, which warranted the development of certified test methodologies by the American Society for Testing and Materials (ASTM).^{5,6}

The use of PVC bags for containing RRS filters contaminated with chloroform is a concern due to the potential reactions of chlorine with PVC monomer. Although the reported reactions have been performed in liquid form (a state not likely to occur in the RRS filter), similar mechanisms leading to swelling and degradation could result in the vapor state. A key parameter to determine is the vapor concentration of chloroform when a contaminated filter is placed and sealed in a PVC bag. The relationship between chloroform loading and vapor concentration in the headspace can be determined with isotherms representing the adsorbent's equilibria with chloroform.

3. VAPOR EQUILIBRIA

The adsorption and desorption of chloroform from the RRS filter will be largely influenced by the ratio of the vapor and adsorbed phase concentrations. At equilibrium, any change in the concentration of the vapor and adsorbed phases is characterized by the isotherm. The desorption of chloroform from the RRS filter will result when the vapor phase concentration decreases below an equilibrium point. In an effort to maintain equilibrium as governed by the isotherm, chloroform will desorb from the adsorbent's surface into the vapor phase. Similarly, an RRS filter (exposed to chloroform and placed into a clean bag and sealed) will attempt to reach equilibrium by desorbing chloroform into the headspace. The resultant vapor concentration will depend on the initial loading of chloroform on the adsorbent and the volume (free-space) contained by the filter bag.

As stated above, the vapor phase concentration can be estimated from initial loadings of chloroform on the adsorbent using an expression for the adsorption equilibria. The concentration above the adsorbent bed can be represented by a single isotherm expression for each temperature condition. The shape of the adsorption-desorption isotherm is typically Langmuir and has the basic form:⁷

$$Q = \frac{bc}{1 + bc} \quad (1)$$

The linear portion of the isotherm can more simply be represented by $Q = kc + b$, where Q is the adsorbed phase concentration per weight of adsorbent (g/g), k is the slope of the linear region of the isotherm, Q versus C (g/g per g/L), b is the intercept of the linear region with Q (g/g), and c is the concentration of chloroform in the vapor phase

(g/L). The mass of chloroform, m , is a constant and at equilibrium it will be distributed between the adsorbed phase and the vapor phase determined by the isotherm expression Q . Therefore, the mass balance can be represented by:

$$m = CV + QM \quad (2)$$

$$= CV + M(kC + b) \quad (3)$$

where, m equals the total mass of chloroform in grams (g), M is the total mass of adsorbent in grams (g), C is the concentration of the vapor phase, and V is the volume of headspace (L). The vapor phase concentration for a given chloroform loading Q and headspace volume V is determined by rearrangement of (3):

$$C = \frac{m - bM}{V + kM} \quad (4)$$

Equation 4 can be used to estimate the headspace concentration of chloroform inside a PVC bag containing the contaminated RRS filters.

4. EXPERIMENTAL METHOD

The first part was to determine the equilibrium concentrations of chloroform in the gas and solid phase at 25 and 55 °C over a range that calculated the loadings of the RRS filter in actual use. The final part of the investigation determined the compatibility of PVC when exposed to chloroform vapors by measuring the permeation and weight pickup of chloroform.

4.1 Adsorption Isotherms.

Adsorption isotherms were measured for chloroform (Fischer Optima grade) on Ionex activated carbon. The adsorbent was a coconut base material sized to 8-16 mesh, with a bulk density of 0.439 g/mL. Isotherms were measured at 25 and 55 °C, representing a temperature range of the filters in storage. Concentrations of chloroform exposed to the adsorbent ranged from 0.10 to 5.0 mg/L and represented the range anticipated in a typical RRS operation.¹ The apparatus used to measure the equilibrium values consisted of a microbalance (CAHN-200) to determine weight pickup, a gas cell infrared (IR) analyzer (MIRAN IA) to measure the headspace concentrations, and a stainless steel bellows pump for circulation of the gas phase (Figure 1). The volume of the adsorption system was 7.0 L. The total system volume was 7.0 L. Incremental injections of chloroform (approximately 2.0 μ L) were added to the headspace and circulated across the sample. Equilibrium was determined when there was no further change in the vapor phase concentration and weight pick-up of the carbon sample.

4.2 Desorption from Preloaded Adsorbent.

Chloroform desorption equilibria were measured on samples previously equilibrated between 0.05 and 0.28 g/g. Chloroform was added by vapor deposition to carbon samples in a sealed glass container. The desorption test chamber used was a finite volume system similar in design to the adsorption isotherm apparatus, less the microbalance (Figure 2). Vapor phase concentrations were measured with the IR analyzer. Three gram samples of adsorbent containing chloroform were placed into a glass test tube supported on a fritted quartz disk. A pump in series with the closed loop system was adjusted to allow 250 mL/min of air to circulate. The total headspace volume was 6.5 L. The test system was thermally jacketed to maintain the temperature at 25 and 55 °C. Equilibrium was determined when there was no further change in the vapor phase concentration.

4.3 PVC-Chloroform Compatibility.

Chloroform absorption measurements were obtained on (samples of 10 mil PVC) using the closed system microbalance described in Figure 1. The concentration of chloroform was 10.0 mg/L. This level of chloroform concentration was selected to represent the upper end of the highest concentration obtained in the RRS operation. Chloroform vapor was circulated across a sample of PVC suspended from the microbalance and supported by a wire basket. The temperature was maintained at 25 °C. The sample of PVC was allowed to equilibrate in the test system before adding the chloroform. The diluent was ambient air with a 50% relative humidity (RH). The weight increase of the PVC sample in the presence of chloroform was recorded and represented the uptake of vapor into the PVC sheet.

Permeation measurements were obtained on samples of PVC by exposing chloroform vapors to one side of the sheet. Sections of PVC were cut from whole filter bags and placed in a test apparatus similar in design to the desorption system. The area of the PVC swatch was 9.8 cm². On one side of the swatch, a 500-mL chamber was fitted with a septa port and was used to contain the chloroform vapors. The concentration of chloroform was selected at 1.0, 10.0, and 100 mg/L. On the other side of the PVC sheet, a second chamber was used to circulate air through the IR detector and then back into the chamber (Figure 3). If penetration of chloroform vapor occurred, the concentration of the mixture with air would be detected. The penetration of chloroform vapor was tested at 25 and 55 °C and was evaluated over a 3- to 10-day period.

5. RESULTS AND DISCUSSION

5.1 Chloroform Equilibria.

The adsorption of chloroform on Ionex adsorbent (a coconut carbon) is highly favorable and produced a shape of the basic Langmuir type with no desorption hysteresis present (Figure 4). As anticipated, there is a significant difference in the adsorption capacity with temperature. A three-fold increase in vapor pressure results by increasing the

temperature from 25 to 55 °C.⁸ At the elevated temperature, the adsorption capacity decreases 50-75% of the capacity at 25 °C. On samples preloaded with chloroform, the amount desorbed increased in proportion to the loading and to the temperature of the bed (Figure 5). The desorption equilibrium values are in good agreement with the adsorption isotherms, indicating that the primary sites of adsorption are in the micropores with no significant hysteresis.

Analytical expressions (equation 4) for the isotherms were approximated by segmenting the adsorption curves into two linear regions to obtain estimates of the vapor concentration at loadings of 0.1 and 0.5 g/g. The expressions were used to estimate the vapor concentration inside of the RRS filter sealed by a PVC bag (Tables 1-4). At 55 °C, an RRS filter with 0.1 g/g loading would contain a total mass of 1786.5 g of chloroform. Depending upon the headspace volume (1.0 to 50.0 L), 0.7 to 38.1 g of chloroform could desorb into the headspace and produce a vapor concentration of approximately 0.7 mg/L. Ratios of headspace volume to bed volume indicated little change in the vapor phase concentration. This was primarily due to the large quantity of adsorbent (and adsorbate) present in the relatively small headspace volume. Further analysis showed that a significant change in the vapor phase would result if the amount of adsorbent was reduced by several orders of magnitude.

Similarly, an increase in chloroform loading to 0.5 g/g at 55 °C would produce approximately 13.0 mg/L of vapor in the headspace. The significant increase in vapor concentration required to obtain 0.5 g/g loading was due to a reduction in the slope of the isotherm beyond 0.1 g/g. In practice, this estimate was probably less realistic, because actual RRS operations would not allow the filter to be exposed to this high of a loading without prior knowledge of significant breakthrough of chloroform at the effluent of the filter. At 0.5 g/g of chloroform, 8932 g of chloroform would be contained on the RRS filter which is equivalent to 6 L of adsorbed liquid.

5.2 PVC-Chloroform Compatibility.

The results of the PVC permeation test showed no penetration of chloroform over a 3- to 10-day period when exposed to 1.0 and 10.0 mg/L of vapor at 25 and 55 °C. A third exposure level at 100 mg/L was performed also with no apparent penetration of chloroform. Inspection of the PVC sheet following exposure did not show any visible deterioration, swelling or discoloration.

Further analysis by a microbalance showed that PVC absorbed chloroform to 0.28% of its original weight over a 12-hr period when exposed to a 10.0 mg/L vapor concentration. The uptake corresponds to the 33.6 µg of chloroform per square centimeter of PVC.

Although it was shown that the effect of chloroform vapor on PVC was negligible, it is well documented that PVC and liquid chloroform are incompatible.⁴ The interaction during liquid contact, while being more severe than vapor, may accelerate the degradation. A similar process may occur with vapors as evidenced in the short-term vapor absorption measurements. Qualitatively, over the 90-day storage life (allowed for

contaminated filters) permeation of chloroform would not be likely, but initial stages of degradation may occur.

6. CONCLUSIONS AND RECOMMENDATIONS

The desorption of chloroform vapors from the Rapid Response System filter can result in concentrations of approximately 0.2 to 13.0 mg/L when sealed in a closed container. The amount of desorption is primarily dependent upon the initial loading of chloroform on the adsorbent and the temperature of the filter bed.

At vapor concentrations of 10.0 mg/L, the equilibrated weight uptake on polyvinyl chloride (PVC) sheet was $33.6 \mu\text{g}/\text{cm}^2$, which was achieved over a 12-hr period. The results support the work of other investigators that showed significant uptake of chloroform on PVC in liquid immersion tests.⁴

Although no permeation of chloroform through PVC sheet was observed during the 10-day exposure periods, the fact that chloroform absorbs on PVC sheet could indicate the initial stages of long term permeation. Considering the filters are double-bagged and have a 90-day storage limit, it is unlikely that chloroform will permeate the barriers. However, as an additional safeguard, it is recommended that other materials compatible with chloroform be used in the future.

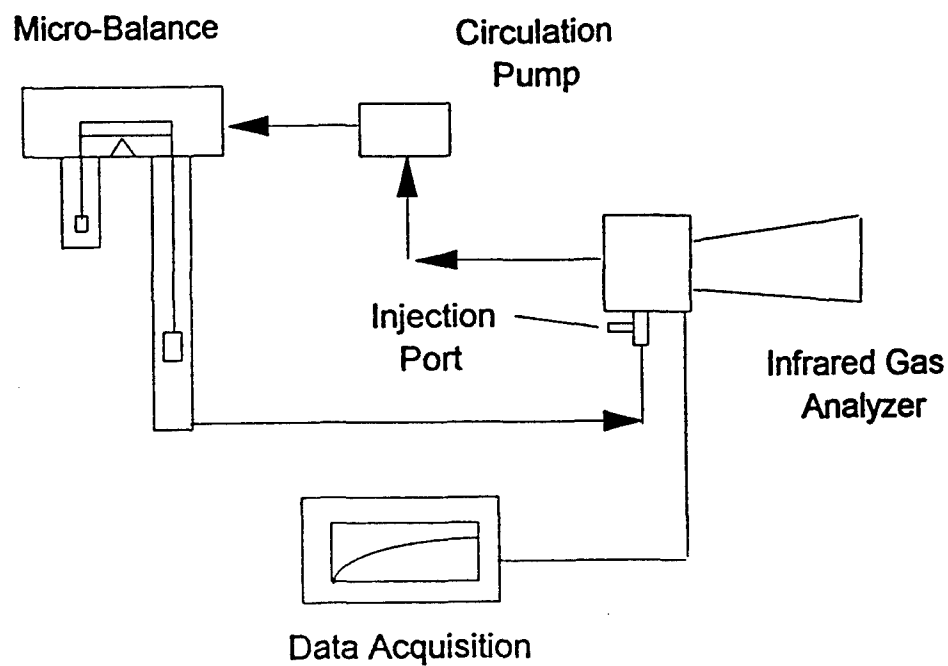


Figure 1. Schematic of Adsorption Isotherm Apparatus

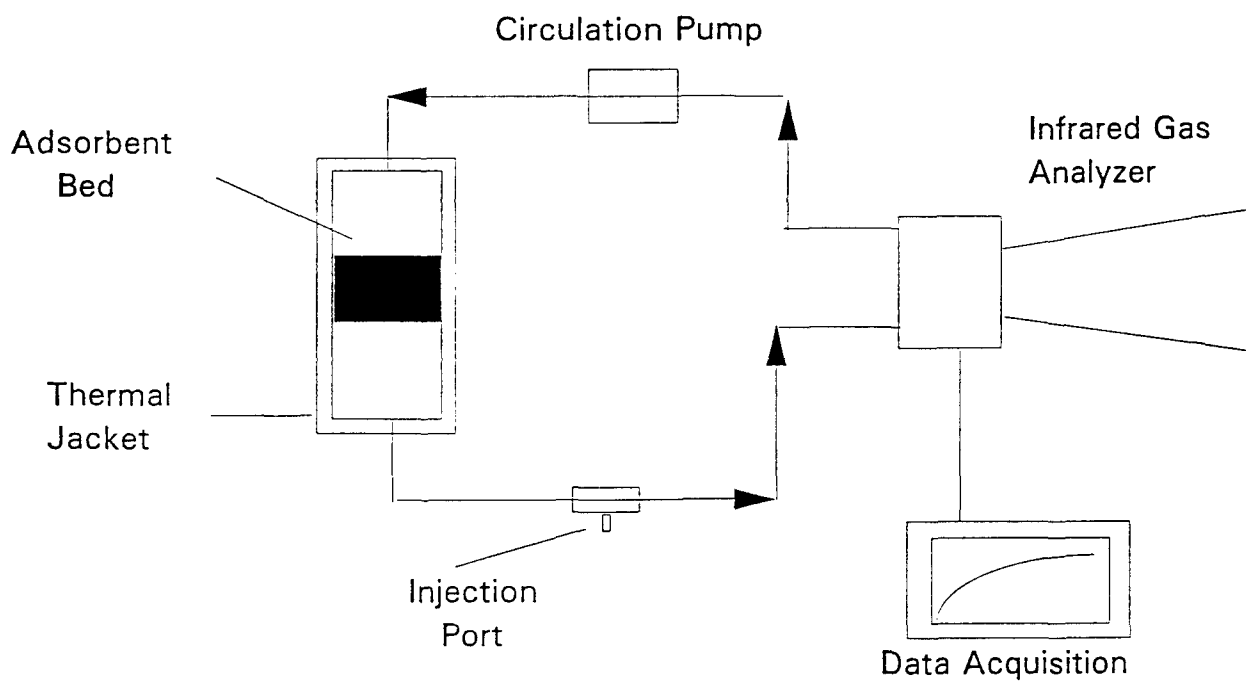


Figure 2. Schematic of Desorption Apparatus

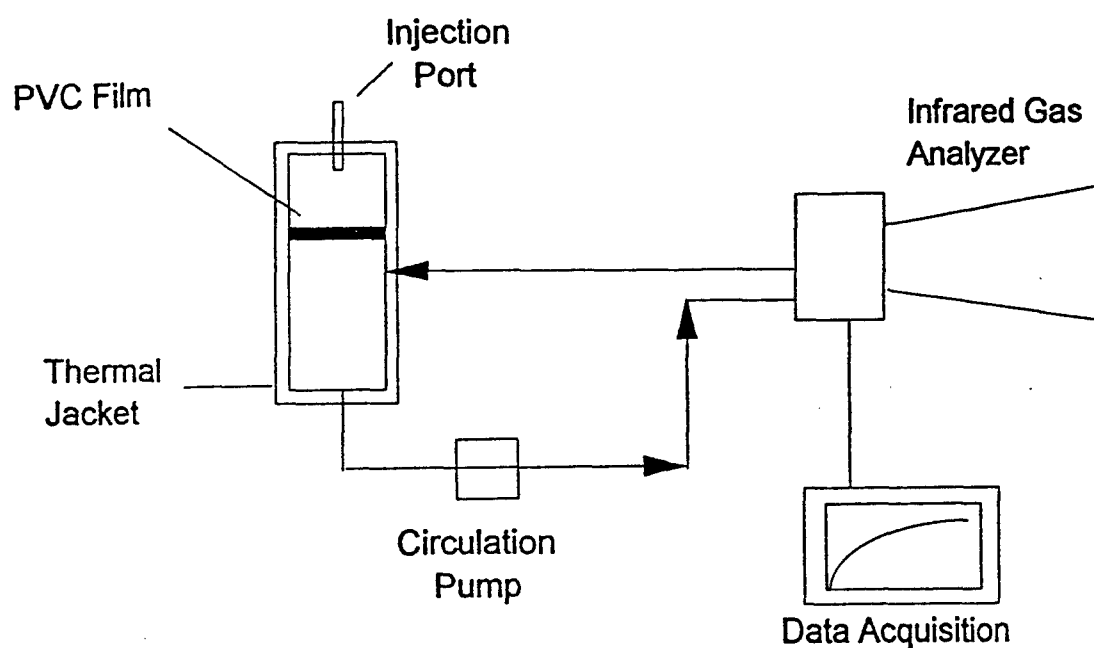


Figure 3. Schematic of PVC-Chloroform Permeation Test Apparatus

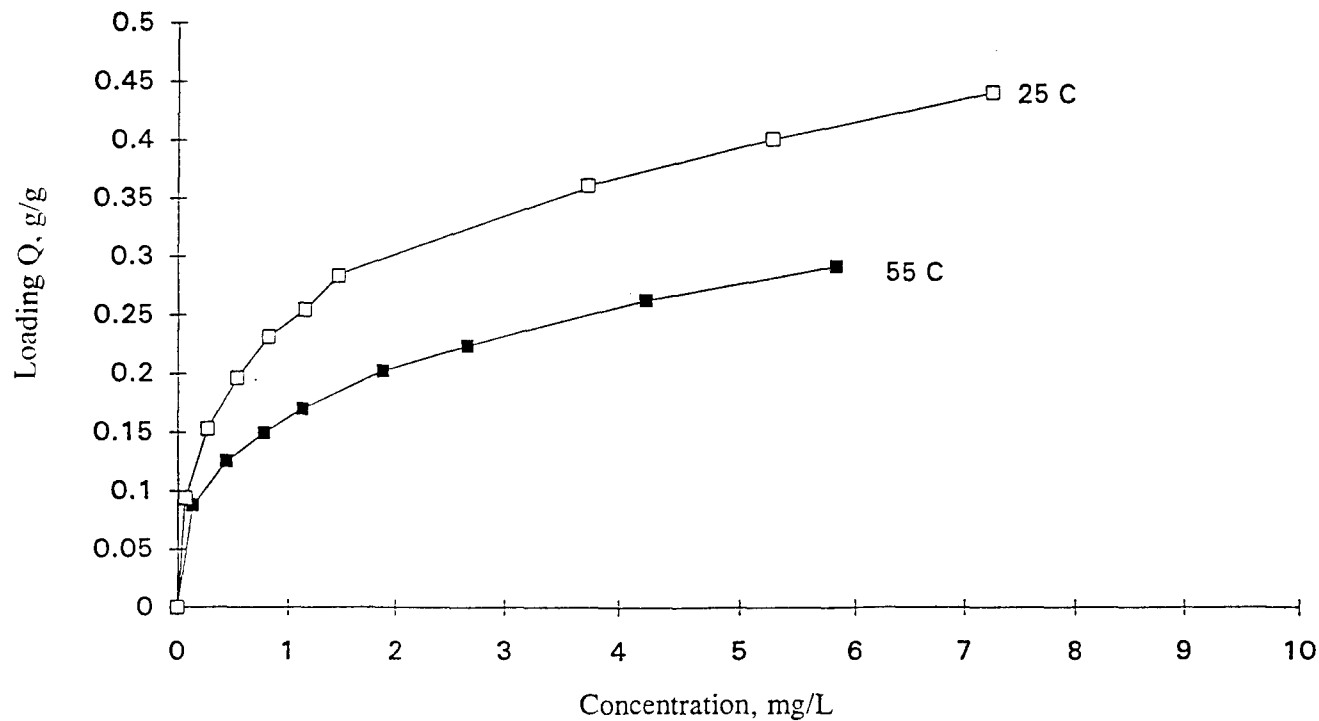


Figure 4. Adsorption Isotherms of Chloroform and Coconut Carbon at 25 and 55 °C

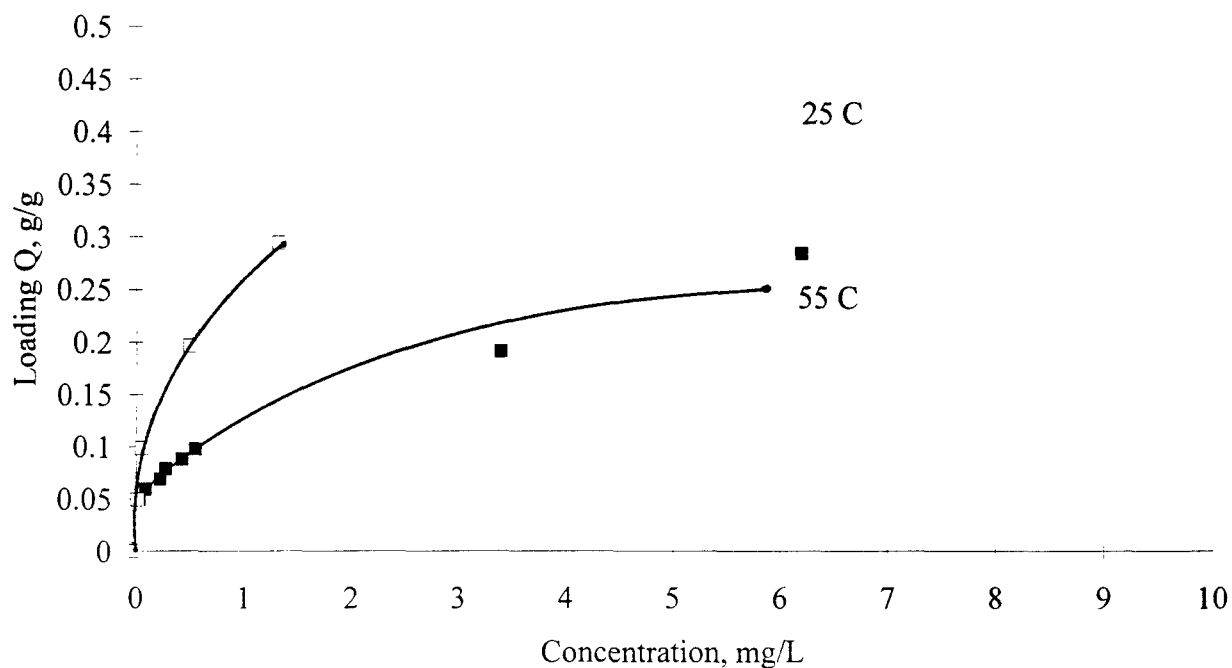


Figure 5. Desorption Isotherms from Samples of Coconut Carbon Preloaded with Chloroform at 25 and 55 °C

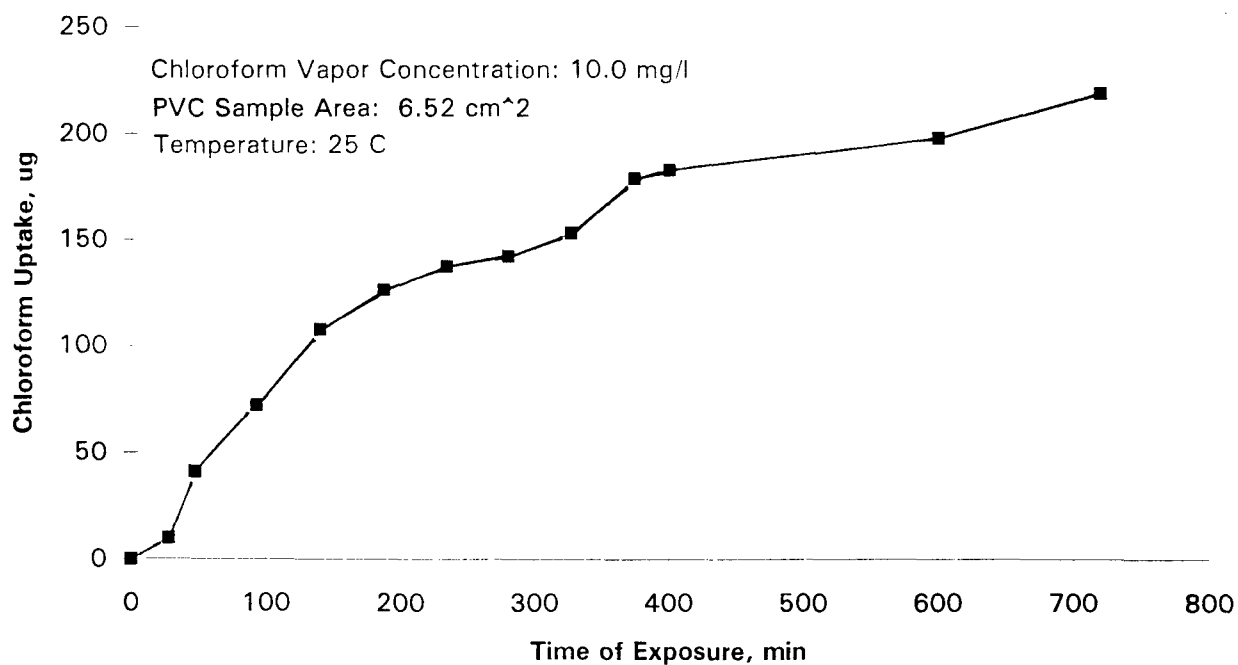


Figure 6. Uptake of Chloroform on PVC Sheet at 10.0 mg/L Vapor Concentration (Ambient Temperature: 25 °C)

Table 1. Estimate of Chloroform Headspace Concentrations in the RRS Filter
(Chloroform Loading: 0.1 g/g, Temperature: 25 °C)

Headspace Volume (L)	Bed Volume (L)	HS:Bed Ratio	Bed Mass (g)	Loading (g/g)	Total Chloroform (g)	Final Concentration (g/L)	Headspace Mass (g)
1	39.7	0.025	17865	0.1	1786.5	0.0002505	0.00025
10	39.7	0.25	17865	0.1	1786.5	0.0002505	0.00250
20	39.7	0.50	17865	0.1	1786.5	0.0002505	0.00501
30	39.7	0.75	17865	0.1	1786.5	0.0002505	0.00751
40	39.7	1.00	17865	0.1	1786.5	0.0002505	0.0100
50	39.7	1.25	17865	0.1	1786.5	0.0002505	0.0125

Table 2. Estimate of Chloroform Headspace Concentrations in the RRS Filter
(Chloroform Loading: 0.5 g/g, Temperature: 25 °C)

Headspace Volume (L)	Bed Volume (L)	HS:Bed Ratio	Bed Mass (g)	Loading (g/g)	Total Chloroform (g)	Final Concentration (g/L)	Headspace Mass (g)
1	39.7	0.025	17865	0.5	8932.5	0.00826	0.0083
10	39.7	0.25	17865	0.5	8932.5	0.00826	0.0825
20	39.7	0.50	17865	0.5	8932.5	0.00826	0.1652
30	39.7	0.75	17865	0.5	8932.5	0.00826	0.2478
40	39.7	1.00	17865	0.5	8932.5	0.00826	0.3303
50	39.7	1.25	17865	0.5	8932.5	0.00826	0.4130

Table 3. Estimate of Chloroform Headspace Concentrations in the RRS Filter
(Chloroform Loading: 0.1 g/g, Temperature: 55 °C)

Headspace Volume (L)	Bed Volume (L)	HS:Bed Ratio	Bed Mass (g)	Loading (g/g)	Total Chloroform (g)	Final Concentration (g/L)	Headspace Mass (g)
1	39.7	0.025	17865	0.1	1786.5	0.000762	0.000762
10	39.7	0.252	17865	0.1	1786.5	0.000762	0.00762
20	39.7	0.504	17865	0.1	1786.5	0.000762	0.0152
30	39.7	0.756	17865	0.1	1786.5	0.000762	0.0229
40	39.7	1.01	17865	0.1	1786.5	0.000762	0.0305
50	39.7	1.26	17865	0.1	1786.5	0.000762	0.0381

Table 4. Estimate of Chloroform Headspace Concentrations in the RRS Filter
(Chloroform Loading: 0.5 g/g, Temperature: 55 °C)

Headspace Volume (L)	Bed Volume (L)	HS:Bed Ratio	Bed Mass (g)	Loading (g/g)	Total Chloroform (g)	Final Concentration (g/L)	Headspace Mass (g)
1	39.7	0.025	17865	0.5	8932.5	0.0130	0.0130
10	39.7	0.252	17865	0.5	8932.5	0.0130	0.1296
20	39.7	0.504	17865	0.5	8932.5	0.0130	0.2591
30	39.7	0.756	17865	0.5	8932.5	0.0130	0.3887
40	39.7	1.01	17865	0.5	8932.5	0.0130	0.5183
50	39.7	1.26	17865	0.5	8932.5	0.0130	0.6478

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